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# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Improvements relating to the manufacture of High Concentrated Hydrogen Peroxide Solutions by Rectification of Diluted Solutions

We, SOCIETE DES PRODUITS PEROXYDES, a company organized under the laws of France, of 75, Quai d'Orsay, Paris (VIIe), France, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :—

This invention relates to the manufacture of high concentrated hydrogen peroxide solutions by rectification of diluted solutions, and more particularly hydrogen peroxide up to 90% concentration for example, and substantially free from impurities.

As it is known, hydrogen peroxide produced by different processes, as for example from persulfuric acid or persulfates, is dilute and contains varied impurities, and in order to get it pure and concentrated, hydrogen peroxide obtained from the above mentioned raw materials is vaporized, this vaporization being followed either by one or more rectifications, or by a total or fractional condensation.

For the rectification or rectifications, an apparatus is used which includes one or more fractionation columns, in the lower part of which enter the hydrogen peroxide vapors issuing from a separator of impurities. Each column is topped with a condenser and its bottom is made up with a boiler. If highly concentrated hydrogen peroxide is needed, the liquid running out from the bottom of the column undergoes one or several similar treatments.

In another embodiment of these processes, the liquid to be concentrated is vaporized in a climbing film evaporator, but this disposition, though offering the advantage that the boiler contains only a small amount of highly concentrated liquid in a thin layer and without liquid storage, suffers the drawback that a continuous running of the apparatus is not easily attainable without elaborate devices or permanent watching, the whole liquid to be vaporized being in the

form of an irregular film which climbs in one or more vertical tubes whence result peaks in the output of hydrogen peroxide vapors.

This invention overcomes more particularly this drawback and applies to the known processes in which the boiler receives the liquid to be concentrated in its upper part and delivers it in its lower part. As in the above mentioned processes of vaporization by a climbing film, the liquid amount in operation is small.

According to the invention, in a rectification column giving up high concentrated, for example 90%, hydrogen peroxide, the vaporization of the high boiling fraction (i.e. the high concentrated H.P.), in the bottom of said column, is carried out in at least two superposed pans through which the fraction flows in series, the upper pan of which is fed by the high boiling liquid fraction, the total amount of this liquid, distributed between all the pans, being just sufficient to deliver the vapors for the rectification and the high concentrated hydrogen peroxide obtained being continuously withdrawn without any storage in the vaporizer.

Under such conditions, the vaporization is achieved only on the smallest necessary liquid amount divided between several heating devices and is effected on more and more concentrated liquids, the last vaporization being consequently carried up on the most concentrated liquid but in a small amount. Thus are much lessened the hazards of decomposition of high concentrated hydrogen peroxide that occur when vaporization is achieved on a large amount of liquid in the boiler. Heat expense is of course rather greater than it would be in this last case, but safety is much increased.

Besides, according to another mode of carrying out the invention, calorific yield may be highly increased if a rectification of

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ascending vapors on the successive pans is simultaneously achieved. This may be done for example by interposing rectification plates between the pans or through heated  
5 rectification plates, for example heated hollow plates with inside heating and with a horizontal, substantially plane area where-upon the liquid flows in a thin film, being thus simultaneously rectified and vaporized.  
10 So is increased the percentage difference between the liquid contained in the first vaporization pan and the high concentrated liquid contained in the last vaporization pan, what improves the safety degree above  
15 mentioned.

The above mentioned boiler is topped with a rectification column that is fed with hydrogen peroxide vapors, for example 35% vapors, introduced into the rectification  
20 column. They arise from the vaporization of hydrogen peroxide 35% which is followed with an elimination of impurities. In the column portion which is over the feeding point, ascending vapors are poorer and  
25 poorer in hydrogen peroxide and are condensed by direct contact with the tubes of the upper water-cooled dephlegmator. The vapors made up only with water and volatile impurities escape from the upper part of the  
30 dephlegmator. Falling down through the rectification column, the liquid becomes concentrated in hydrogen peroxide and falls into the above mentioned boiler. In order to avoid any substantial storage of the  
35 liquid, the bottom of the boiler is cone-shaped and ends with a pipe out of which is continuously extracted the hydrogen peroxide that is immediately cooled.

Extracting devices may be set up at  
40 different heights of the vaporizer in order to pick up liquids of various percentages in hydrogen peroxide.

A liquid for adjusting pH at any desired value, and any known stabilizer may be  
45 introduced in the rectifying column.

In above mentioned conditions, the running of the distilling and concentrating apparatus is quite stable. After having set up the output of the hydrogen peroxide to be  
50 concentrated in the rectifying column, it is sufficient to adjust the water feed of the dephlegmator and the heating of the boiler. Then is collected at the bottom of the boiler concentrated, pure and stable hydrogen  
55 peroxide with a constant titer. This titer is adjusted by the water feed in the dephlegmator, a fall of the titer resulting from an increase of the water feed, and conversely. Should the titer of the hydrogen peroxide  
60 which collects in the bottom of the vaporizer tend to drop through an accidental reason, the temperature of the liquid falls whence results an increased vaporization which tends to raise the concentration of

hydrogen peroxide. Should the vapours  
65 entering the dephlegmator hold a greater percentage of hydrogen peroxide, the condensation temperature arises and a greater liquid amount tends to be condensed, thus restoring the previous concentration.  
70

The accompanying drawing shows an example of an embodiment of the invention in the case when a vaporization and a rectification are simultaneously carried out, by means of rectifying plates with inside  
75 heating.

Raw hydrogen peroxide, with for example a concentration of 35%, is sucked through the pipe 1, and vaporized in a coil 2, which is set inside a box 3, heated by steam entering  
80 at 4 and issuing at 5. Hydrogen peroxide goes through a liquid trap 6 and by pipe 7 goes into the rectification column, provided either with Raschig rings or with any other device having the same purpose, as bubbling  
85 plates either with bubble caps or perforated. This column is provided in its upper part with a condenser 8 of any kind, intended to deliver the necessary reflux and with a tube 9, out of which the non-condensed  
90 vapours issue.

At the lower part of the column is located the vaporizing device. In the shown example, it comprises three hollow pans, the upper parts of which are in the form of  
95 rectifying plates provided as usual with bubble caps and overflows. The interior of the pans is traversed by a flow of steam, going in by pipes 10 and out by pipes 11. The overflows with which the plates are  
100 provided are so set as to keep on each plate a shallow liquid layer. These plates act as the usual rectifying plates, the liquid being rectified thereon at the same time as it is vaporized.  
105

As it has been said above, the vaporizer is cone-shaped at its lower part, and provided with a discharge cock 12.

HAVING NOW particularly described and ascertained the nature of our said invention  
110 and in what manner the same is to be performed, we declare that what we claim is:—

1. Improved process of manufacture of high concentrated, for example 90%, hydrogen peroxide solutions by rectification  
115 of diluted solutions, in which the high boiling liquid fraction resulting from the rectification is vaporized in at least two superposed pans through which the fraction flows in series, the upper pan of which is fed  
120 by the high boiling liquid fraction, the total amount of this liquid, distributed between all the pans, being just sufficient to deliver the vapors for the rectification and the high concentrated hydrogen peroxide obtained  
125 being continuously withdrawn without any storage in the vaporizer.

2. Embodiment of the process according

to claim 1, wherein the vapor issuing from the liquid on each pan is rectified when passing from a pan to the next upper one.

3. Embodiment of the process according to claims 1 and 2, the hydrogen peroxide obtained being withdrawn at different heights of the vaporizer.

Dated this 11th day of July, 1949.  
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